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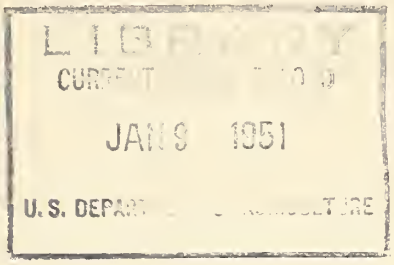

RECOVERY OF NICOTINE FROM *NICOTIANA RUSTICA* BY STEAM DISTILLATION

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CONTENTS

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INTRODUCTION

The demand for nicotine during recurring periods in the past has exceeded the supply available from the wastes of the tobacco industry, such as stems, damaged leaves and sweepings. In the course of the Eastern Regional Research Laboratory's investigation of *Nicotiana rustica* as a commercial source of nicotine, studies were made of methods of recovering nicotine from this plant. This paper reports research to determine if the commercial method of distilling nicotine from tobacco could be used with green and dried *N. rustica*.

EQUIPMENT

Figure 1 shows a diagrammatic sketch of the equipment. The distillation vessel was a conventional, cylindrical, agitated drier. It was horizontal, 2 feet in diameter, and 4 feet long; all parts were steam jacketed, including the charging and discharging door. It was equipped with a ribbon-type agitator attached to a horizontal, steam-heated shaft, which rotated at 6 r.p.m. Vapor evolved from the drier was cleaned by passing it through a flannel filter bag, which was also steam jacketed. The dust-free vapor was condensed in a water-cooled, surface condenser. In some of the experiments, nicotine was scrubbed from the condenser vent gas by a dilute hydrochloric acid solution in a 2-inch diameter column packed to a height of 4 feet with 3/8-inch Raschig rings.

OPERATION

Green *N. Rustica*

The first group of experiments was made with leaves and whole plants (leaves and stalks) of green *N. rustica*. The material was cut with a rotary knife cutter and disintegrated in a hammer mill equipped with a 1/2-inch screen. Approximately 200 pounds of the material thus disintegrated was put in the drier, lime was added to release nicotine, and the two materials were mixed by the agitator. In some of the experiments, the lime and disintegrated material were mixed before they were put in the drier. Steam at 100 p.s.i. gage was admitted to the heating jackets of the drier, and distillation was

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* Report of a study made under the Research and Marketing Act of 1946.

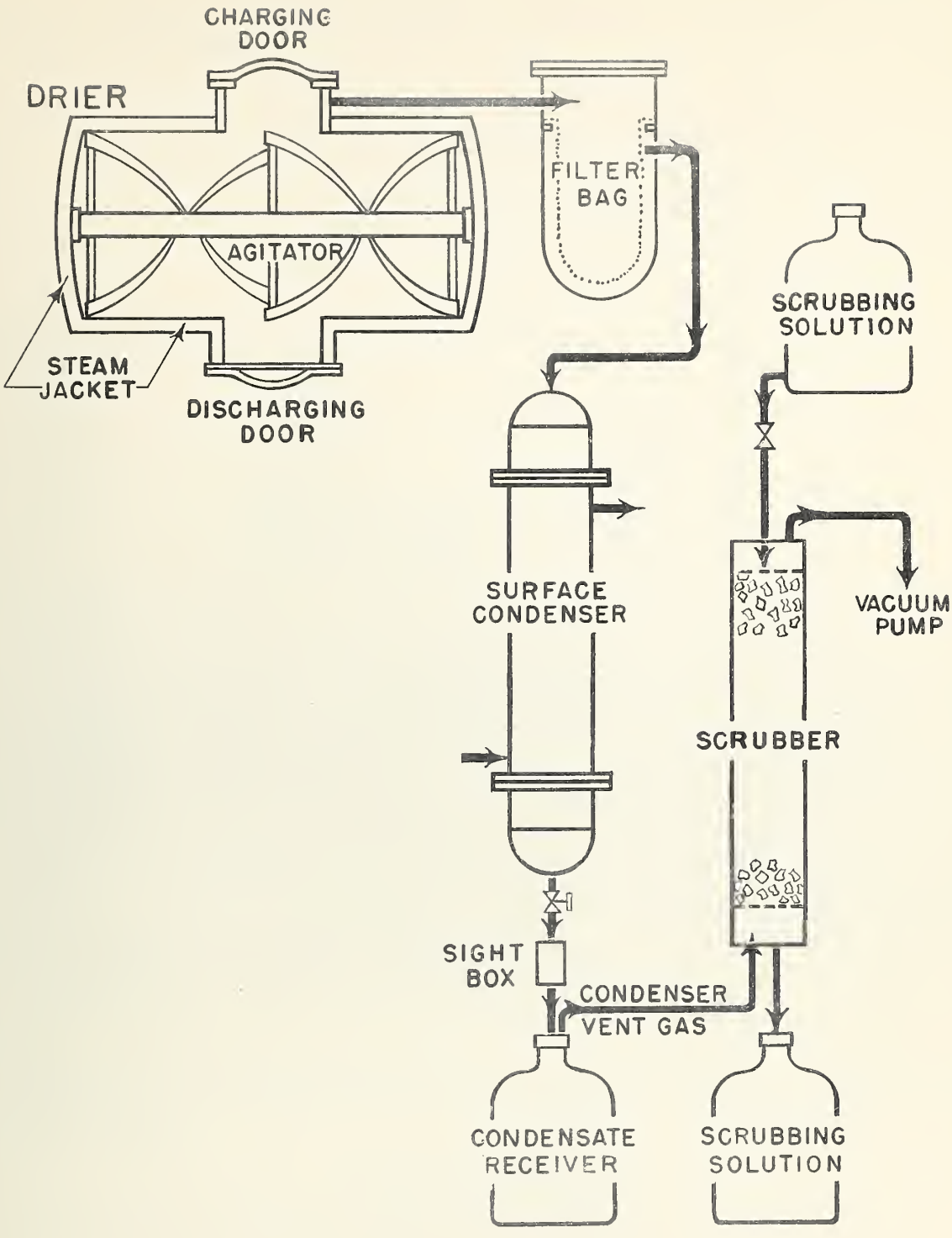

FIG. 1--DIAGRAMATIC FLOW SHEET.

carried to dryness. Experiments were made with distillation at atmospheric pressure, at vacuum and with several combinations. The different types of distillation are tabulated in Table I.

An attempt was made to obtain a nicotine balance, that is, to have the amount of nicotine in the products equal the amount of nicotine in the charge. With green *N. rustica*, the balance was erratic, however, since the hammer milling produced a soupy mixture of liquid and solid phases, which was difficult to sample accurately. Nicotine was determined by the official method of the A.O.A.C., in which nicotine is precipitated by silicotungstic acid.

The lime used throughout this work was a high calcium, hydrated lime; the following analysis was supplied by the shipper:

	Percent
$Ca(OH)_2$	95.62
MgO	0.80
Through 100 mesh	100
Through 200 mesh	99.5
Through 325 mesh	99.0

Dried *N. Rustica*

In a second group of experiments, dried (air-cured) plant material was used. The moisture content was approximately 12 percent. The dried material, which comprised leaves, stalks and whole plants, was ground in a hammer mill equipped with a 1/4-inch screen. It was sampled with a seed-sampling device. Since a representative sample could be obtained on the dried ground product, nicotine balances were satisfactory. *N. rustica*, lime and water were put in the drier, and the mixture was distilled to dryness at atmospheric pressure. In some of the experiments only part of the water was added with the lime; distillation was started, and water was added continuously as it was distilled. The drier did not have provisions for admitting stripping steam, and the necessary connection could not be made, so the continuous addition of water was used as a substitute for stripping steam.

Tobacco

The tobacco used in these experiments was a typical blend of stems from cigarette and cigar factories. It was processed in the same manner as the dried *N. rustica*.

DISCUSSION OF RESULTS

Table I and Figure 2 give results.

Green *N. Rustica*

Leaves and whole plants: A series of experiments (1 to 6 in Table I) was made to determine nicotine recovery, when leaves and whole plants of green

TABLE I - SUMMARY OF RESULTS

Expt. No.	Part of plant[1]	Lime used per 100 pounds of plants	Type of distillation[2]	MOISTURE CONTENT		NICOTINE CONTENT				NICOTINE BALANCE - % OF TOTAL			
										Recovered in distillate			
				Of plants	After drying	Of plants	Of total distillate	Of residue (MFB)	Lost during drying	atmos. fraction	Vacuum fraction at end	Left in residue	Total accounted for
--	--	lbs.	--	%	%	%	%	%	%	%	%	%	%
						GREEN *N. RUSTICA*							
1	W	10	1	89.31	--	5.33	.53	.067	--	85.0	--	2.3	87.3
2	W	10	1	86.89	--	5.87	.77	.10	--	91.5	--	2.7	94.2
3	W	10	1	87.92	--	6.79	.81	.12	--	85.7	--	3.4	89.1
4	L	10	1	89.46	--	5.98	.62	.10	--	89.3	--	2.6	91.9
5	L	10	1	85.03	--	2.74	.39	.057	--	80.4	--	3.0	83.4
6	L	10	1	89.23	--	4.18	.51	.072	--	101.6	--	3.1	104.7
7	L	10	2	89.51	--	3.24	.19	.36	--	48.8	--	25.4	74.2
8	W	10	3	87.40	75.8	3.97	.92	.07	2.9	78.5	2.2	3.1	86.7
9	W	10	3	87.69	63.0	3.82	1.47	.12	5.5	72.3	5.0	5.6	88.4
10	W	10	4	83.41	56.0	3.07	1.92	.18	2.3	79.3	5.3	9.2	96.0
11	W	10	3	85.48	52.6	5.79	2.24	.52	3.7	53.8	1.5	14.6	73.8
12	W	10	5	88.78	43.0	4.28	2.26	.13	1.7	59.9	4.7	5.6	71.9
13	W	1-1/4	6	84.7	--	4.09	.67	.26	--	90.8	2.2	6.6	99.6
14	W	2-1/2	6	84.6	--	3.25	.63	.15	--	107.8	.9	5.3	114.0
15	W	5	6	85.1	--	4.09	.70	.23	--	98.4	1.6	6.6	106.6
16	W	1-1/4	6	85.3	--	2.95	.52	.58	--	92.4	9.2	20.7	122.3
17	W	2-1/2	6	87.4	--	3.11	.50	.48	--	99.6	9.1	18.0	126.7

DRIED *N. RUSTICA*

18	L	80	7	87	--	7.57	1.05	.33	--	89.8	2.1	8.0	99.9
19	L	80	7	92	--	7.19	.62	.26	--	100.3	1.4	6.9	108.6
20	L	80	8	40	--	7.71	.77	.099	--	91.1	1.3	2.5	94.9
21	S	20	8	40	--	1.88	.63	.02	--	96.8	2.5	1.3	100.6
22	W	20	8	40	--	4.80	.80	.032	--	95.5	0.2	2.0	97.7

TOBACCO

23	TW	12	7	55.5	--	1.23	.86	.02	--	90.6	1.2	1.7	93.5
24	TW	12	7	35.0	--	1.15	1.53	.13	--	75.7	6.7	12.1	94.5
25	TW	12	9	13.9	--	1.26	1.62	.05	--	72.0	0.8	4.2	77.0

[1] *W = Whole plant (minus roots); L = leaves only; S = stalks only; TW = trade wastes.*

[2]

Type	*Water added at start*	*Drying stage*	*Distillation stage*	*Water added during distillation*	*Finish of distillation*
1	*None*	*None*	*Atmos.*	*None*	*Atmos*
2	*do*	*do*	*Vac.(28 in.)*	*do*	*Vac.(28 in.)*
3	*do*	*Atmos.*	*Atmos.*	*do*	*do*
4	*do*	*Vac.(28 in.)*	*do*	*do*	*do*
5	*do*	*do*	*do*	*Twice, stepwise*	*do*
6	*do*	*None*	*do*	*None*	*do*
7	*All*	*do*	*do*	*do*	*do*
8	*Part*	*do*	*do*	*Continuously*	*do*
9	*do*	*do*	*do*	*3 times, stepwise*	*do*

N. rustica were distilled with lime at atmospheric pressure. These experiments showed that essentially the same results were obtained with either leaves or whole plants. About 3 percent of the total nicotine was left in the residue from either material; the remaining 97 percent should have been recovered in the distillate. The nicotine recovered, however, ranged from 80 to 102 percent, a variation which can be attributed to the difficulty of obtaining a representative sample of the starting material. Concentration of nicotine in the distillate ranged from 0.39 to 0.81 percent. No significance should be attached to this variation, since in all cases sufficient water was present to steam distill the nicotine. Furthermore, essentially all the water and all the nicotine were distilled by carrying the distillation to dryness, and thus the concentration of nicotine in the distillate was controlled by the ratio of nicotine to water in the original material. Recovery of nicotine from the residue would be impracticable. Experiments showed that so much water would be required for this distillation that the concentration of nicotine in the distillate would be only a few hundredths of 1 percent.

Vacuum and atmospheric distillation: Another series of experiments (5 to 7 in Table I) was made to compare the effect of vacuum distillation and atmospheric distillation on the recovery of nicotine. Pertinent data are summarized in Table II.

Table II. Comparison of Vacuum Distillation and Atmospheric Distillation

Type of Distillation	Vac. 28 inches	Atmos.
Experiment No.	7	5 and 6, Ave.
Nicotine left in residue, % in Plants	25.4	3.0
Nicotine recovered in distillate, % in Plants	48.8	91.0
Nicotine accounted for, % in Plants	74.2	94.0
Nicotine in distillate, %	0.19	0.45

The explanation of these results can be seen from curves A and B of Figure 2. In vacuum distillation (curve A), the first fraction was low in nicotine and the last was high, with the result that the water was exhausted before the nicotine was distilled and the nicotine then decomposed. Thus if one ignores the sampling errors inherent in succulent material, 25.4 percent of the total nicotine was left in the residue and an additional 25.8 percent of the nicotine decomposed, leaving only 48.8 percent of the nicotine recovered in the distillate. In atmospheric distillation (curve B, experiment 5), the first fractions were higher in nicotine than the last, and as a result essentially all the nicotine was distilled before the water was exhausted. Thus with atmospheric distillation, only 3 percent of the total nicotine was left in the residue, 91 percent was recovered in the distillate, and the remaining 6 percent was within the experimental limit of accurate sampling.

Results of this general character were anticipated because of the fact that the vapor pressure of nicotine falls off more rapidly with increase in temperature than does the vapor pressure of water.

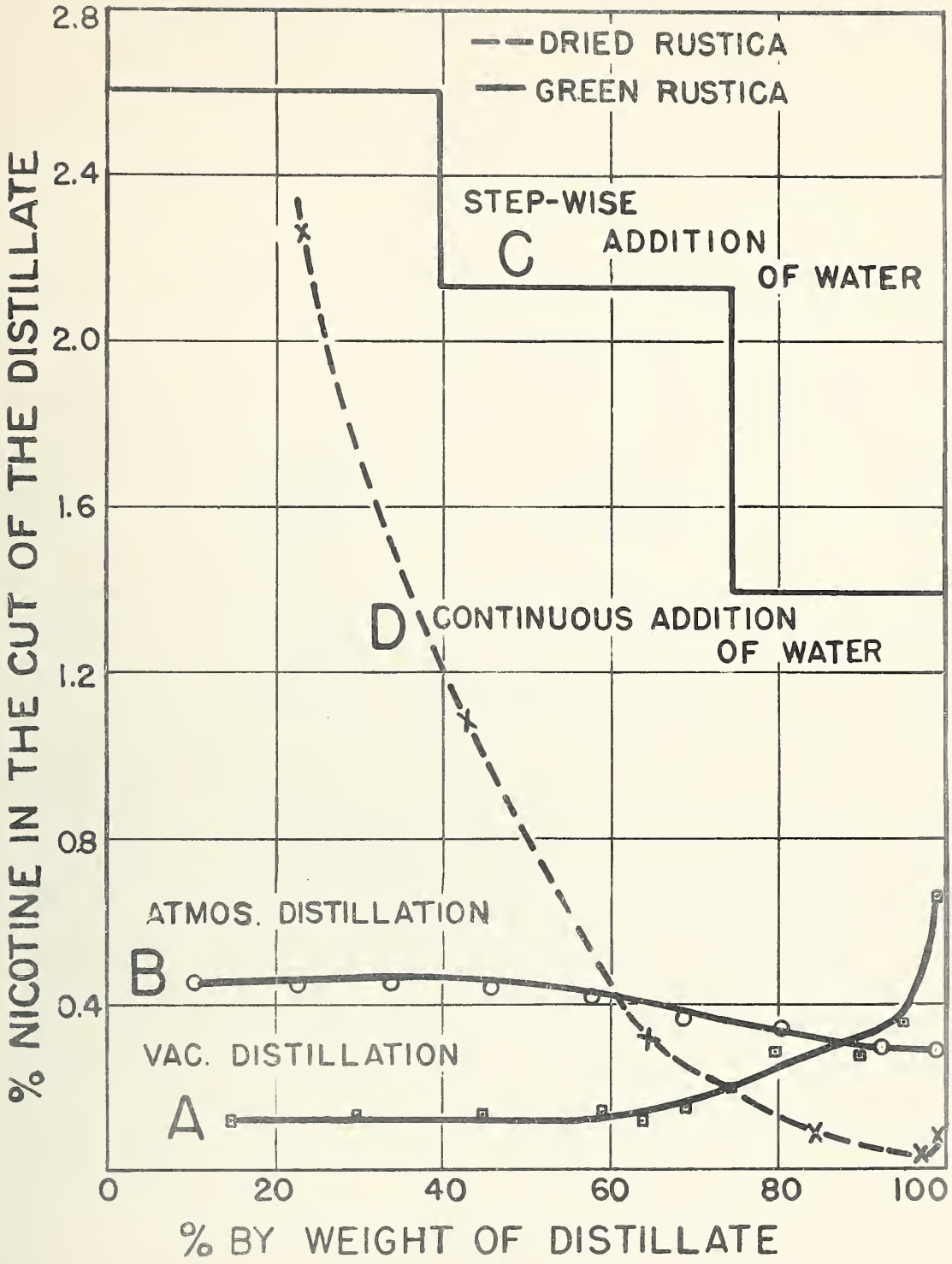

FIG. 2--CONCENTRATION OF NICOTINE IN VARIOUS CUTS OF THE DISTILLATE.

Effect of moisture: Experiments 8 to 11 comprise a series designed to obtain maximum concentration of nicotine in the distillate, combined with maximum recovery of nicotine, by varying the moisture content of the plant material. In these experiments, the moisture content was reduced by drying the material in the still operated as a drier, lime was added later, and then distillation was carried to dryness at atmospheric pressure. Drying was done both at atmospheric pressure and at a vacuum of 28 inches. Nicotine distilled during the drying stage averaged 4 percent of the total with atmospheric drying and 2 percent with vacuum drying. Pertinent data are summarized in Table III.

The average concentration of nicotine in the distillate during drying was 0.03 percent, a value so low that it would not warrant recovery of nicotine. Hence, nicotine evolved during the drying stage is considered nonrecoverable.

TABLE III EFFECT OF MOISTURE CONTENT ON NICOTINE RECOVERY

Expt. No.	Moisture Content After Drying %	Nicotine in Distillate %	Nicotine Left in Residue- of Total %
6	[1]89.2	0.51	3.1
8	75.8	0.92	3.1
9	63.0	1.47	5.6
10	56.0	1.92	9.2
11	52.6	2.24	14.6

[1] *Not dried*

Table III shows that the combination of maximum concentration of nicotine in the distillate and maximum nicotine recovery was not obtained simultaneously. The optimum moisture content for the combined objectives would be about 63 percent (experiment 9). The steam required for processing material of this moisture content would be approximately 67 pounds per pound of nicotine distilled and, assuming that steam costs 75 cents per 1000 pounds, the cost of the steam would be about 5 cents. In experiment 6, in which the nicotine content of the material was about the same as that in experiment 9, the steam consumed would be approximately 195 pounds, which would cost 14.6 cents per pound of nicotine distilled. Thus, drying to 63 percent moisture would effect a saving of 9.6 cents per pound of nicotine distilled. The drying, however, would have to be done without using the drier, for example, by field wilting, since there would be no saving of steam, if all the drying were done in the drier.

Although atmospheric distillation was used in experiments 6 and 8 to 11, it was found preferable to finish the distillation at a vacuum of 28 inches. Finishing at vacuum not only eliminated the obnoxious fumes when the drier was opened but it also increased recovery of nicotine by 2 or 3 percent.

This increase in nicotine recovery under vacuum near the end of the run, as compared with atmospheric operation can probably be attributed to recondensation of nicotine in the still. The recondensation would be less under vacuum.

Stepwise addition of water during distillation: Since data in Table III showed that nicotine concentration in the distillate could be increased by keeping the moisture content of the charge to a minimum, experiment 12 was made to see if this could be done without significant reduction in nicotine recovery, if water were added stepwise. In this experiment, the green material was dried to 43 percent moisture, lime was added, and the distillation was carried to dryness. Water was added twice to bring the moisture content to 14 percent; distillation was carried to dryness after each addition of water. The results are summarized in Table IV.

TABLE IV EFFECT ON NICOTINE RECOVERY OF ADDING WATER STEPWISE

	% of Total Nicotine
Lost in drying stage	1.7
Left in final residue	5.6
Recovered in distillate	64.6
Total accounted for	71.9

The concentration of nicotine in the distillate was high, 2.26 percent, and the nicotine left in the residue was reasonably low, 5.6 percent; however, only 71.9 percent of the nicotine was accounted for. This discrepancy is far in excess of what can be accounted for by sampling errors. There was undoubtedly some destruction of nicotine resulting from repeated heating of the plant material. Concentration of nicotine in the various fractions of the distillate from experiment 12 is shown in curve C of Figure 2.

Effect of lime: In the foregoing experiments, in which 10 pounds of lime was used per 100 pounds of wet material, a baked deposit formed on the inner surface of the still. Its thickness, about 3/8-inch, corresponded to the clearance between the agitator blades and the inner wall. The deposit was obviously undesirable, since it greatly reduced transfer of heat. In experiments 13 to 17 (Table I) different amounts of lime were used in an attempt to avoid sticking. Table V shows the results.

The best results were obtained by incorporating the lime into the green material outside the still. The lime cannot be effectively incorporated in the still because, since the material is later to be heated, there is a relatively large clearance between the agitator blades and the wall. Almost any type of paddle or ribbon-type mixer which has close clearance between the blades and wall can be used for incorporating the lime. The largest amount of lime incorporated outside the still was 2-1/2 pounds per 100 pounds of green material. This produced no sticking; however, the nicotine left in the residue was 5.3 percent. It is possible that use of more lime, incorporated outside the still, would release more nicotine.

TABLE V EFFECT OF LIME ON STICKING

Expt. No.	Pounds Lime per 100 Pounds of Plants	Where Incorporated	Sticking	% of Total Nicotine Left in Residue
15	5	In still	Severe	6.6
17	2-1/2	do	Severe but localized	18.0
16	1-1/4	do	do	20.7
13	1-1/4	Outside	Moderate	6.6
14	2-1/2	do	None	5.3

Dried *N. Rustica*

Since one of the systems for using *N. rustica* as a commercial source of nicotine might entail drying the plant before processing, some experiments (18 through 22, Table I) were made with dried plant material. The green plants were dried in unheated air to about 12 percent moisture. In experiment 18, sufficient water was added to the dried, ground plants to produce moisture corresponding to that in green *N. rustica*, for example, 87 percent. Ten pounds of lime was then added per 100 pounds of wet material, and the mixture was distilled. The residue after distillation contained 8 percent of the total nicotine. In the green plants under similar conditions, there was about 3 percent. Apparently it is more difficult to distill the nicotine from dried plant tissue than from green material. This was further confirmed in experiment 19, in which a still larger quantity of water was added. Therefore, to obtain more effective recovery of nicotine, water was added (in experiments 20, 21 and 22) continuously during the process. It was necessary to add water instead of steam, since we had no facilities for adding stripping steam to the drier, as is done industrially. In all cases, recovery of nicotine was good, and less than 3 percent of the total nicotine remained in the residue. Curve D of Figure 2 shows the concentration of nicotine in the distillate at various stages of the distillation in experiment 22.

Since, in experiment 20 as well as in experiments 18 and 19, there was some sticking on the inner wall of the still, the amount of lime used in experiments 21 and 22 was reduced from 80 pounds per 100 pounds of dried material to 20 pounds. This reduction eliminated sticking and did not impair the recovery. In distilling dried material, the lime and water could be incorporated in the still.

Tobacco

Experiment 23 was made to compare the performance of the experimental unit with that of stills used commercially for obtaining nicotine from tobacco wastes. Table VI shows the comparison.

TABLE VI COMPARISON OF RESULTS WITH EXPERIMENTAL AND INDUSTRIAL STILLS

	Experimental Unit	Typical Industrial Distillation
Nicotine in plants, %[1]	1.23	1 to 2
Nicotine in distillate, %	0.86	0.55 to 0.65
Nicotine in residue, % of total	1.7	3 to 5
Water in plants, %	55.5	25 to 30
Stripping medium	None	Steam
Lime used, lbs./100 lbs. tobacco	12	10 to 15
Steam pressure in jacket, lbs./sq.in.ga.	100	50 to 70
Drier pressure during distillation	Atmos.	Atmos.
Drier pressure at end	Vac.	Vac.

[1] *Moisture-free basis.*

The data in Table VI show that our experimental unit performed at least as well as the typical commercial unit. Both the concentration of nicotine in the distillate and the removal of nicotine from the tobacco were somewhat superior in the experimental unit.

The concentration of nicotine in the distillate is, of course, a function of the quantity of water or steam introduced into the material with respect to the nicotine in the starting material. In the experimental unit, the quantity of steam introduced (water was actually used) was probably less than the stripping steam plus water used industrially. To lessen the water or steam introduced is desirable, as it increases the concentration of nicotine in the distillate. The use of water, instead of injected steam, generates the steam *in situ*, thereby achieving more effective stripping of the nicotine. Similar results could presumably be achieved with steam if the equipment were designed to obtain thorough mixing of steam and material.

SUMMARY AND CONCLUSIONS

This work demonstrated that nicotine can be obtained from either green or dried *N. rustica* by distillation in the same general type of equipment commercially employed for distilling tobacco waste. When the green plants were distilled at atmospheric pressure, less than 5 percent of the nicotine was left in the residue, and the nicotine in the distillate was about 0.8 percent. In commercial distillation of tobacco waste, 3 to 5 percent of the nicotine is left in the residue, and the distillate contains about 0.6 percent.

Steam distillation of nicotine at vacuum is not suitable, since about 25 percent of the nicotine is left in the residue and an additional 25 percent is decomposed.

Nicotine balance was erratic with green *N. rustica* but was reasonably good with dried *N. rustica* or tobacco.

When all the water for volatilizing the nicotine was added to dried *N. rustica* at the start of the distillation, about 8 percent of the nicotine was left in the residue; however, this was reduced to 2 percent by adding water continuously during the distillation.

In distilling *N. rustica,* sticking in the still was eliminated by using 2-1/2 pounds of lime per 100 pounds of material; however, the lime must be thoroughly incorporated in the material outside the still. With dried *N. rustica,* sticking did not occur when 20 pounds of lime was added per 100 pounds, and the two materials could be mixed in the still. In the industrial extraction of tobacco waste, 10 to 15 pounds of lime per 100 of tobacco is used.

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